

Amendments to the Specification:

Please insert the following paragraph beginning on page 1, line 5:

This application claims the benefit of International Patent Application No. PCT/IB2003/005612 filed September 24, 2003, which claims priority of Belgian Patent Application No. 2002/0557, filed September 25, 2002.

Please replace the paragraph beginning on page 1, line 6 with the following amended paragraph:

BACKGROUND

~~The present invention concerns oxidised~~ Oxidised zinc compounds in the form of powder with improved pourability, the method of manufacturing these and their use in the field of polymers, in particular elastomers, are disclosed.

Please replace the paragraph beginning on page 1, line 16 with the following amended paragraph:

Zinc oxide is used in ~~very varied~~ various fields, such as the fields of enamels, varistors, oils, paints, various reagents for polymers, both plastics and elastomers, and even in animal feeds. For these many applications, it is in various forms, generally in the form of a powder. In the specific field of elastomers, zinc oxide is employed in particular as a sequestering catalyst for the vulcanisation reaction, during the cross-linking of the elastomers, or as a semi-reinforcing filler:

- as a catalyst for the cross-linking reaction (that is to say the creation of a three-dimensional bridge lattice by virtue of a vulcanisation agent, such as sulphur), the zinc oxide is combined with stearic acid in order to form an activating complex soluble in gum, which combines with the vulcanisation agent to allow the fixing of the macro-molecular chains of the elastomer; the mechanisms of actions are transient (not very stable complexes) and/or ill defined in the case of variations in certain parameters (type of zinc oxide, specific surface, etc);

- as fillers, in the same way as other powdery products (carbon black, chalk, kaolins, talc, etc) it confers specific properties on the product (conductivity, improvement in resilience, etc).

Please replace the paragraph beginning on page 4, line 24 with the following amended paragraph:

SUMMARY

~~The inventors surprisingly discovered that, by~~ By using an atomisation process for the drying of the oxidised compounds of zinc, in particular zinc oxide, ~~an atomisation process,~~ and by precisely adjusting certain operating parameters, it is ~~was~~ possible to obtain an oxidised zinc compound with improved pourability and dispersibility, and with good reactivity, without altering its specific surface.

Please replace the paragraph beginning on page 4, line 32 with the following amended paragraph:

~~The present invention therefore concerns an~~ An oxidised zinc compound in the form of micro-spherules, characterised in that it has a Flodex index of less than 15, and preferably less than 10 is disclosed.

Please replace the paragraph beginning on page 5, line 33 with the following amended paragraph:

Preferably, the apparent density of the oxidised zinc compounds in powder form ~~accorded to the invention~~ is greater than approximately 0.8. In addition, they advantageously have a compressibility index $[(\text{packed density} - \text{apparent density}) \times 100 / \text{packed density}]$ of less than approximately 20%, preferably less than approximately 15% and advantageously between approximately 10% and 12%.

Please replace the paragraph beginning on page 6, line 17 with the following amended paragraph:

~~The present invention also concerns a~~ A method of preparing the aforementioned oxidised zinc compound is disclosed, characterised in that it consists of injecting, by ~~means of~~ a nozzle, an aqueous suspension of the said oxidising compound, having a solid matter content of 25% to 70% by weight, and a pressure of 10 to 100 bar approximately, within an atomisation chamber, in a gas flow entering at a temperature of 250° to 800°C approximately and leaving at a temperature of 50° to 300° approximately.

Please replace the paragraph beginning on page 6, line 28 with the following amended paragraph:

The aqueous suspension of oxygenated compound of zinc at 25-70% by weight solid matter, preferably 35% to 60% by weight, is sufficiently fluid to be sprayed (atomised) by ~~means of~~ a nozzle.

Please replace the paragraph beginning on page 7, line 1 with the following amended paragraph:

The drying technique ~~according to the invention~~ allows the addition of certain additives in the suspension and makes it possible to obtain perfect homogenisation within the product. In particular, if it is wished to increase the solid matter content up to 45-70% by weight, it may be advantageous to add a dispersing agent stabilising the said suspension, such as a polyacrylate.

Please replace the paragraph beginning on page 7, line 23 with the following amended paragraph:

The method ~~according to the invention~~, referred to as atomisation, is a drying process which preserves all the physical and chemical properties (in particular the specific surface) of the particle as from the precipitation reaction, whilst modifying the macroscopic structure of the

product by assisting the formation of micro-spherules with a close granulometric distribution. This is because it is clear that the micro-spherules have a D_{50} (size corresponding to 50% of the total granulometric distribution curve) of between approximately 50 and 200 μm , advantageously between approximately 50 and 100 μm , and preferably between approximately 70 and 90 μm .

Please replace the paragraph beginning on page 8, line 14 with the following amended paragraph:

~~In the present invention, in~~ In combination with the adjustment of the pressure, the choice of the diameter of the discharge from the spray nozzle (for example between 0.8 and 6 mm) makes it possible to vary the size of the required micro-spherules, between approximately 50 and 100 μm , always producing them in a narrow granulometric distribution.

Please replace the paragraph beginning on page 8, line 26 with the following amended paragraph:

The oxidising compound obtained by ~~the~~ this method ~~according to the invention~~ no longer has any “clumping” and makes it possible to obtain a flow of the “micro-ball” type, which makes the product very easy to convey through pneumatic channels without clogging the pipes.

Please replace the paragraph beginning on page 8, line 32 with the following amended paragraph:

This is an important advantage in the use of the oxidised zinc compounds ~~according to the invention~~ in industrial mixers with pneumatic migration of the powder ingredients, and for their use in polymer matrices, preventing the formation of invisible agglomerates during the mixing phase.

Please replace the paragraph beginning on page 10, line 3 with the following amended paragraph:

A binder, such as polyol, could nevertheless be added to the oxidised zinc compound in order better to bind the unitary particles to each other, forming the micro-spherule. However, such a binder is not necessary ~~in the invention~~ and makes it possible not to “contaminate” the material in which the micro-spherules will be incorporated (elastomers, etc.).

Please replace the paragraph beginning on page 10, line 11 with the following amended paragraph:

The oxidised zinc compounds ~~according to the present invention~~ disclosed here can be produced by the method described above, finding an advantageous use in the cross-linking of polymers, in particular in the vulcanisation of elastomer.

Please replace the paragraph beginning on page 10, line 20 with the following amended paragraph:

DESCRIPTION OF THE DRAWINGS

~~The present invention will be illustrated by non-limiting~~ Non-limiting examples, with reference to the accompanying figures, are presented in which:

Please replace the paragraph beginning on page 10, line 24 with the following amended paragraph:

Figure 1 is a diagram presenting the device for implementing the method ~~according to the invention,~~

Please replace the paragraph beginning on page 11, line 18 with the following amended paragraph:

DETAILED DESCRIPTION

Example 1 : Device

A device for implementing the method ~~according to the invention~~ presented in Figure 1 comprises a cylindrical atomisation chamber 1 with a conical bottom in which a stream of hot air circulates. The incoming air first passes through a filter 2 and a burner 3 for preheating it and, at a temperature of 550°C, enters the top part of the atomisation chamber 1 through a distributor with fins 4. The air flow during the test was around 700 N m³/h.

Please replace the paragraph beginning on page 12, line 4 with the following amended paragraph:

The particles of ZnO obtained fall into the bottom part of the chamber where they are collected and discharged through the valve 7 in order for example to be bagged immediately. At this level between 85% and 90% of the FF (free flowing) zinc oxide is collected in the form of spherules with a mean diameter between 70 and 100 µm containing less than 0.5% residual water. The finest particles are discharged in the air leaving the chamber through the pipe 8 under the suction action of a fan 9. They are then separated from the emerging air for example in a cyclone 10 and recovered by the valve 11 in order possibly to be recycled in the preparation of the suspension in the disperser 12. Approximately 10% to 15% by weight fines (non agglomerated power in the form of spherules) are thus immediately separated from the zinc oxide spherules constituting the FF ZnO ~~of the invention~~, and recycled.

Please replace the paragraph beginning on page 12, line 35 with the following amended paragraph:

The following Table 1 presents the results of analysis of two series of zinc oxide dried by the atomisation method ~~according to the invention~~ by means of the device described above.

Please replace the paragraph beginning on page 13, line 10 with the following amended paragraph:

These two oxides were put in suspension in water (40%-45%) and were subjected to the described method ~~according to the invention~~ in a device like the one presented in Example 1.

Please replace the paragraph beginning on page 15, line 5 with the following amended paragraph:

Table 2 below, associated with Figures 2a to 2c, makes it possible to compare the physical and chemical characteristics of zinc oxides dried by different methods :

- a. the conventional ZnO CR thermal method
- b. the so-called “spin flash” ZnO HR 30 method
- c. the ZnO FF (Free Flowing) atomisation method ~~according to the invention ZnO FF (Free Flowing)~~ described here
- d. a zinc oxide dried by a “flash” method

Please replace the paragraph beginning on page 17, line 9 with the following amended paragraph:

Figure 2 presents the comparisons of the results of analysis of the images for these three oxides: frequency according to the area in μm^2 of the section of the particles observable on the section. These curves show principally that ~~the ZnO according to the invention (ZnO FF dried by atomisation)~~ ZnO FF dried by atomisation has, after incorporation in the rubber matrix, fewer large particles than the ~~ZnO~~ ZnO CR (dried by a conventional thermal method): in fact a significant reduction in the peak around $0.02 \mu\text{m}^2$ is noted. The profiles of the curves for ZnO FF and SnO HR30 are similar.

Please replace the paragraph beginning on page 18, line 9 with the following amended paragraph:

These results show better resistance to aging of the rubbers containing 3 p and 1.5 p ZnO FF ~~according to the invention.~~

Please replace the paragraph beginning on page 18, line 35 with the following amended paragraph:

It is clear that, with the ZnO FF ~~according to the invention~~ (cf. Figure 6), the reconstitution is appreciably decreased, which avoids the use of additional products with anti-reconstitution action. The activation energy of the ridges created with ZnO FF is therefore greater than that of other types of zinc oxide.

Please replace the paragraph beginning on page 19, line 28 with the following amended paragraph:

It will be noted in Figure 7 that the time of use of the moulds can be doubled by using the free flowing zinc oxide according to the invention described herein, compared with the other zinc oxides tested (HR30, CR, Bayer Aktiv).